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TUNGSTEN ALLOY FABRICATION OF PENETRATOR MATERIALS

August 1981

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FINAL REPORT

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Prepared for

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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Tungsten heavy alloy bars 1.35" in diameter were prepared and evaluated for uniformity of properties. Alloys contained 97.1% to 97.3% tungsten with primary emphasis on the 97.1W-1.6Ni-0.7Fe-0.5Cu-0.1Co composition. To achieve high uniform densities with this alloy, it was necessary to use fine tungsten powder (~1-1.5μm) and slow heating rates between 1000°C and the sintering temperature. This was not necessary with similar alloys containing no copper. Tensile elongation was found to be strongly dependent on

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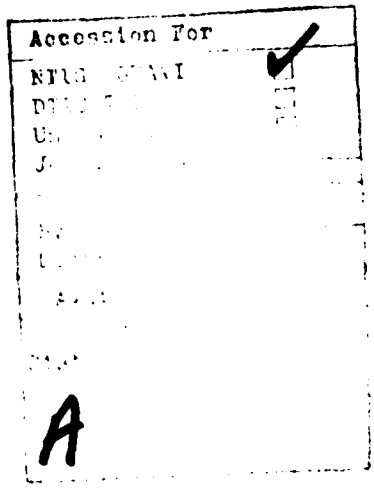
oxygen levels in the sintered bars. Low oxygen levels gave the best results with 16-17% elongation when oxygen levels were below 6 ppm. These very low oxygen levels were only achieved with alloys containing no copper.

PREFACE

This report was prepared under Contract DAAG46-78-C-0009 and administered under the direction of AMMRC with Mr. A. Joseph DeLai as Technical Supervisor.



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INTRODUCTION

In March of 1978 we began work on a contract with Army Materials and Mechanics Research Center to study tungsten heavy alloys. Primarily we were to study the 97.1W-1.6Ni-0.7Fe-0.5Cu-0.1Co alloy. The contract was divided into three overlapping phases. The first was to see how uniformly we could produce bars of the W-Ni-Fe-Cu-Co alloy. We were to check uniformity within a sintering run and among sintering runs. Uniformity was to be defined by several physical and chemical analyses.

In the second phase we were to characterize the variables in the processing of the bars. In the final phase we were to look at variations of the alloys to see how uniformity and properties were affected.

As a prelude to Phase I, we began tests to develop a procedure to achieve high sintered densities. We considered anything above 99.4% dense to be high density. For these tests we drew on our experience with W-Ni-Fe systems. Unfortunately, the addition of copper and cobalt to the alloys made it behave very differently, and it was almost a year before satisfactory densities were achieved.

We then proceeded with our uniformity studies and alloy variations. We found the bars to be uniform but the elongations were very low. Our work was then directed towards improving the tensile properties. Once we achieved this, we again did a uniformity series and looked at alloy variations to complete the contract.

To simplify our reference to the alloys used in this work, we adopted the following nomenclature. A three-digit number is used to represent the tenths of a percent for the iron, copper, and cobalt. Thus, the standard 97.1%W-1.6Ni-0.7Fe-0.5Cu-0.1Co alloy became the 751 alloy. Because the tungsten was held constant at 97.1%, the nickel content can be determined from the balance. The only exception was a standard Sylvania alloy, WN-107. This alloy has 97.3% tungsten with equal parts of iron and nickel.

OBJECTIVES

The objectives as set forth in the scope of the contract are as follows:

1. Evaluate the uniformity of physical properties, mechanical properties, chemical composition, and structure of W-Ni-Fe-Cu-Co alloys. Assess the variation among and within individual bars of the alloy with respect to (1) density, (2) hardness, (3) tensile properties, (4) fracture mode (SEM), (5) interstitials, (6) metal composition, (7) microstructure, and (8) macrodefects (ultrasonic inspection).

2. Characterize the powders and the process used in fabricating the bars used in the uniformity study. Examples of characterization shall include, but are not limited to, (1) tungsten particle size and distribution, (2) blending, (3) sintering schedule and atmosphere, and (4) post-sintering heat treatments.

3. Evaluate different compositions for uniformity using the eight criteria as referenced in the first objective.

CONCLUSIONS

A. For the 751 alloy (97.1W-1.6Ni-0.7Fe-0.5Cu-0.1Co) we concluded the following:

1. The elongations show a strong correlation to oxygen levels with lower oxygen levels giving higher elongations. It appears that for optimum elongations the oxygen must be below about 6 ppm.

2. Sintered density increases with decreasing tungsten particle size. A Fisher Sub-Sieve Size (FSSS) of 1.0 to 1.5 appears to be optimum.

3. During sintering, the bars must be heated slowly (~30 °C per hour) through the 1000 °C to 1400 °C region to develop good tensile properties.

4. The bars must be very slowly cooled (~50 °C per hour) from the sintering temperature to avoid poor tensile properties in the center of large-diameter bars (1.35" diameter).

5. Because of center-to-edge density gradients, it becomes increasingly difficult to sinter large-diameter bars to full density.

6. Sintering in a wet H_2 atmosphere increases porosity and oxygen content with a corresponding decrease in tensile properties.

B. For the W-Ni-Fe system we concluded the following:

1. Good density can be achieved over a wide range of sintering schedules.

2. It doesn't show the tungsten particle size dependence that the 751 alloy does. Coarser particle sizes appear to be slightly better.

3. Low oxygen values and high elongation can easily be achieved.

C. The microstructure of the 751 alloy has more angular tungsten grains than W-Ni-Fe alloys.

D. Oxygen analysis for tungsten heavy alloys must be done with methods that can use large sample pieces. Grinding of the sample causes severe oxidation.

EXPERIMENTAL PROCEDURE

A. Powder Blends

Our powder blends were made using the elemental powders listed in Table I. Also given are some physical and chemical properties of these powders.

Blends were made by first sifting all powders -200 mesh and then blending them in a V-blender for one hour. Table II lists all blends made for the contract.

B. Pressing

All pressing was done in an cold isostatic press generally using a pressure of 30 ksi. Molds were tapped while being filled and were sealed without evacuation.

C. Sintering

Most of the sintering took place in a Brew Model 901 vacuum furnace. This furnace has tungsten mesh elements, tungsten heat shields, and a 12"x12"x12" hot zone. Dry hydrogen with a dew point of less than -25 °C and a flow rate 10 liters per minute was used for our atmosphere. Temperature was measured by a W-5Re/W-26Re thermocouple in a molybdenum protection tube. All tests run in this furnace are listed in Table III.

A few sintering tests were run in a Vacuum Industries furnace. This furnace has tungsten rod elements, molybdenum heat shields, and a 8"x6"x6" hot zone. It also uses a W-5Re/W-26Re thermocouple in a molybdenum protection tube. Tests run in the V.I. furnace and a few other miscellaneous tests are listed in Table IV.

TABLE I

PROPERTIES OF ELEMENTAL POWDERS

Powder Type	FSSS		C	N_2	O_2
	μm	Porosity (%)	(ppm)	(ppm)	(W/O)
Cobalt-African Metals Extra Fine	1.41	0.767	174	<1	0.310
Nickel Inco 123	4.25	0.610	580	3	0.073
Iron - GAF	4.26	0.610	697	40	0.144
Copper MD-151	14.50	0.617	90	4	0.155
Copper MD-301	8.70	0.555	---	2	0.090
Tungsten M-55 237C	5.25	0.632	12	4	0.018
Tungsten M-30 247C	1.85	0.738	23	116	0.102
Tungsten M-30 294C	2.03	0.727	38	77	0.710
Tungsten M-17 789C	1.06	0.732	---	---	---
Tungsten M-17 918C	1.15	0.721	53	---	0.500

TABLE II

HEAVY ALLOY BLENDS

Blend	Composition (W/O) Ni	Fe	Cu	Co	Tungsten Lot		Wt. (kg)
3781	1.6	0.7	0.5	0.1	WA55-237C	MD-151	10.0
3782	1.6	0.7	0.5	0.1	WA55-237C	MD-151 (-325)	5.0
4781	1.35	1.35	-	-	WA55-237C	-	5.0
4782	1.6	0.7	0.5	0.1	WA55-237C	MD-301	5.0
5781	1.9	0.9	-	0.1	WA55-237C	-	5.0
5782	1.8	0.8	0.2	0.1	WA55-237C	MD-301	5.0
5783	1.9	0.7	0.5	-	WA55-237C	MD-301	5.0
6781	1.6	0.7	0.5	0.1	WA55-237C	Cu,Ni,Co co-reduced	2.5
6782	1.6	0.7	0.5	0.1	WA55-237C	Fine Cu	2.0
6783	1.6	0.7	0.5	0.1	WA30-247C	MD-301	2.0
8781	1.6	0.7	0.5	0.1	WA30-247C	MD-301	5.0
9781	1.35	1.35	-	-	WA30-294C	-	2.0
9782	1.6	0.7	0.5	0.1	WA37-227C	MD-301	5.0
10781	1.6	0.7	0.5	0.1	WA30-294C	MD-301	10.0
10782	1.6	0.7	0.5	0.1	WA30-294C	MD-301	15.0
10783	1.6	0.7	0.5	0.1	WA30-294C	MD-301	15.0
1791	1.6	0.7	0.5	0.1	WA17-789C	MD-301	12.0
2791	1.6	0.7	0.5	0.1	WA17-918C	MD-301	12.0
3791	1.9	0.9	-	0.1	WA17-918C	MD-301	2.5
3792	1.8	0.8	0.2	0.1	WA17-918C	MD-301	2.5
3793	1.7	0.7	0.5	-	WA17-918C	MD-301	2.5
3794	1.6	0.7	0.5	0.1	WA17-918C	MD-301	12.0
4791	1.6	0.7	0.5	0.1	WA17-918C	MD-301	15.0
4792	1.6	0.7	0.5	0.1	WA17-918C	MD-301	6.0*
6791	1.9	0.9	-	0.1	WA17-918C	MD-301	2.5
6792	1.8	0.8	0.2	0.1	WA17-918C	MD-301	2.5
6793	1.7	0.7	0.5	-	WA17-918C	MD-301	2.5
6794	1.6	0.7	0.5	0.1	WA17-918C	MD-301	8.0
8791	1.6	0.7	0.5	0.1	WA17-918C	MD-301	24.0
12791	1.6	0.7	0.5	0.1	WA17-918C	MD-301	24.0
1801	1.9	0.9	-	0.1	WA55-237C	-	12.0
2802	1.6	0.7	0.5	0.1	WA17-918C	MD-301	12.0
2803	1.6	0.7	0.5	0.1	WA17-918C	MD-301	24.0
3801	1.6	0.7	0.5	0.1	WA17-918C	MD-301	12.0

*Tungsten powder Wellexed 3 minutes (high-intensity blender).

TABLE III

SINTERING TESTS IN BREW FURNACE

Test	Ramp 1 (mv/hr)	Hold 1 Temp (°C)	Hold 1 Time (min)	Ramp 2 (mv/hr)	Hold 2 Temp (°C)	Hold 2 Time (min)	Ramp 3 (mv/hr)	Hold 3 Temp (°C)	Hold 3 Time (min)	Cool (mv/hr)	Comments
5	20	1000	30	8	1400	30	5	1580	120	30	
6	20	1000	30	8	1400	30	5	1650	120	30	
7	20	1000	5	20	1400	15	10	1650	180	30	
8	20	1000	5	20	1400	15	10	1650	180	30	
9	16	900	30	3	1400	10	3	1650	240	30	
10	20	1000	5	20	1400	15	10	1650	180	30	
11	20	1000	5	20	1400	15	10	1600	240	30	
12	10	630	60	20	1400	15	10	1600	240	30	
13	20	1000	5	20	1400	15	10	1600	240	10	
14	10	630	60	15	1000	60	45	1600	240	30	
15	20	1000	5	20	1400	15	10	1600	240	30	
20	20	1000	5	20	1400	15	10	1600	120	30	
24	20	1000	5	20	1400	15	10	1600	120	30	
25	20	1000	5	20	1400	15	10	1550	120	30	
26	20	1000	5	20	1400	15	10	1500	120	30	
27	20	1000	5	20	1400	30	1	1500	120	30	
28	20	1000	1	20	1400	1	20	1500	120	30	
30	20	1000	5	20	1400	15				30	
31	20	1000	5	20	1400	15	10	1500	120	30	
33	20	1000	5	20	1400	15	10	1500	120	30	
34	20	1000	60	20	1400	15	10	1500	120	5	Switch to N_2 after 15 minutes in Hold 1

TABLE III (Cont.)

SINTERING TESTS IN BREW FURNACE

Test	Ramp 1 (mv/hr)	Hold 1 Temp (°C)	Hold 1 Time (min)	Ramp 2 (mv/hr)	Hold 2 Temp (°C)	Hold 2 Time (min)	Ramp 3 (mv/hr)	Hold 3 Temp (°C)	Hold 3 Time (min)	Cool (mv/hr)	Comments
35	20	1000	5	20	1400	15	10	1500	120	1	
36	20	1000	60	20	1400	15	10	1500	120	30	
37	20	1000	5	20	1400	15	10	1500	120	30	
39	20	1000	15	20	1400	15	10	1500	120	30	
40	6	900	120	2	1400	60	1	1480	90	2	
41	20	1000	15	20	1400	15	10	1500	120	30	
42	20	1000	15	20	1400	15	10	1500	120	30	
43	20	1000	15	20	1400	15	10	1500	120	30	
44	20	1000	15	20	1400	15	10	1500	120	30	
45	20	1000	15	20	1400	15	10	1500	120	30	
46	5	1000	30	5	1400	15	10	1545	120	30	
47	5	1000	30	5	1400	15	10	1530	120	30	
48	5	1000	30	2	1400	15	10	1520	120	30	
49	5	1000	30	5	1400	15	5	1510	120	30	
50	5	1000	30	5	1400	15	5	1510	120	30	
51	5	1000	30	5	1400	15	5	1510	120	1	
52	5	1000	30	5	1400	15	5	1555	120	30	
53	5	1000	30	5	1400	15	5	1520	120	1	
54	5	1000	30	20	1400	15	10	1525	120	30	
55	5	1000	30	5	1400	15	5	1500	120	30	
56	20	1000	30	5	1400	15	5	1550	120	1	Double H_2 flow
57	20	1000	30	5	1400	15	5	1525	120	1	
58	5	1000	30	5	1400	15	5	1525	120	1	Dew point -30 °C
59	20	1000	30	5	1400	15	5	1520	120	1	
60	5	1000	30	5	1400	15	5	1530	120	1	

TABLE III (Cont.)

SINTERING TESTS IN BREW FURNACE

Test	Ramp 1 (mv/hr)	Hold 1 Temp (°C)	Hold 1 Time (min)	Ramp 2 (mv/hr)	Hold 2 Temp (°C)	Hold 2 Time (min)	Ramp 3 (mv/hr)	Hold 3 Temp (°C)	Hold 3 Time (min)	Cool (mv/hr)	Comments
61	5	1000	30	5	1400	15	5	1530	120	1	
62	5	1000	30	5	1400	15	5	1520	120	1	
63	5	1000	30	5	1400	15	5	1520	120	15	
64	5	1000	30	5	1400	15	5	1515	120	1	Dew point -20 °C
65	5	1000	30	5	1400	15	5	1525	120	1	Dew point 0 °C
66	5	1000	30	5	1400	15	5	1520	120	1	Dew point +20 °C
67	5	1000	30	5	1400	15	5	1520	120	30	
68	5	1000	30	5	1400	15	5	1520	120	1	
69	5	1000	30	5	1400	15	5	1515	120	1	

TABLE IV

SINTERING TEST NOT RUN IN BREW FURNACE

Test 16 - V.I. Furnace - H_2 and Vacuum

15 minutes to 900 °C - hold 1 hour in H_2
Cool to 600 °C and evacuate
20 minutes to 1580 °C - hold 4 hours

Test 17 - No. 3 Muffle Furnace - H_2 Atmosphere

1-hour stoke into hot zone
Hold 4 hours at 1585 °C

Test 18 - No. 3 Muffle Furnace - H_2 Atmosphere

3-hour stoke into hot zone
Hold 4 hours at 1585 °C

Test 19 - Same as Test 18

Test 21 - Small Induction Furnace - H_2 Atmosphere

3 hours at 1560 °C

Test 22 - No. 6 Muffle Furnace - Wet H_2 Atmosphere

2 hours into hot zone
Hold 4 hours at 1585 °C

Test 23 - V.I. Furnace - H_2 Atmosphere

30 minutes to 1000 °C - hold 15 minutes
15 minutes to 1400 °C - hold 30 minutes
15 minutes to 1490 °C - hold 5 minutes

Test 29 - No. 3 Muffle Furnace - H_2 Atmosphere

1¼-hour stoke into hot zone
Hold 2 hours at 1500 °C

Test 32 - V.I. Furnace - H_2 Atmosphere

30 minutes to 1000 °C - hold 15 minutes
15 minutes to 1400 °C - hold 30 minutes
15 minutes to 1500 °C - hold 2 hours

TABLE IV (Cont.)

SINTERING TEST NOT RUN IN BREW FURNACE

Test 38 - V.I. Furnace - H_2 Atmosphere

15 minutes to 1000 °C - hold 5 minutes
15 minutes to 1400 °C - hold 15 minutes
30 minutes to 1500 °C - hold 2½ hours

D. Heat Treating

Most of the heat-treating work was done in the V.I. furnace in vacuum at 1200 °C for 2 hours (Table V). A few tests were done in a tube furnace using a nitrogen atmosphere.

E. Tensile Tests

Tensile tests were made using threaded tensile bars with a 0.25" diameter x 1.25" reduced section. The gauge section was polished circumferentially with 4/0 polishing papers. Cross-head speed was 0.05" per minute to yield and then 0.005" per minute to failure. Elongation was determined by piecing the broken tensile bars together and measuring a 1"-gauge length that had been scribed on the bar. These results are in Table V.

F. Oxygen Analysis

Oxygen determinations were made by Luvak Inc. of Boylston, Massachusetts. They use a vacuum fusion instrument with an iron bath at 1650 °C.

Because extraction time is several minutes, relatively large chunks of material can be used. We initially tried to use our Leco TC30 inert-gas fusion instrument. Maximum operating temperature is 3000 °C and uses a nickel flux with a 40-second extraction time in helium. Because of the short extraction time, smaller pieces must be used. In the comminution of the samples we found that oxidation occurred giving us erratic results. Oxygen results are contained in Table V along with the tensile results.

G. Density

Density determinations were made by water immersion. We believe our standard deviation to be <0.007 g/cc for individual determinations.

TABLE V

ROOM TEMPERATURE TENSILE PROPERTIES AND OXYGEN VALUES

Test	Bar	Alloy	Sample Location	Atm.	Anneal Hours	°C	Elongation (%)	Yield (ksi)	UTS (ksi)	0_2 (ppm)
39	10783A	751	MR	N_2	2	1200	7	82.9	110.1	29
	10783	751	MR		None		0	77.4	78.5	35
40	10782A	751	MR	N_2	2	1000	2	82.5	82.5	
	10782A	751	MR	N_2	2	1000	4	81.6	105.6	
41	1791A	751	MR	N_2	2	1000	1	76.6	83.5	
	1791A	751	MR	N_2	2	1000	3	81.8	96.0	
	10782B	751	MR	N_2	2	1000	Broke out of gauge length			
	10782B	751	MR	N_2	2	1000	6	81.1	104.9	
42	2791	751	MR	N_2	2	1000	5	80.1	96.4	
43	2791A	751	MR				5	84.5	101.8	59
	2791A	751	MR				3	82.9	100.4	
	3794A	751	MR				5	83.5	101.0	
	3794A	751	MR				3	81.8	99.0	
	3794B	751	MR				4	80.9	97.7	55
	3794B	751	MR				4	84.8	103.3	
	3791	901	MR				4	88.5	108.9	62
	3791	901	MR				3	88.4	105.2	60
44	3794C	751	MR	Vac	2	1200	4	83.1	99.1	
	3794C	751	MR	Vac	2	1200	3	81.3	97.9	54
	3794D	751	MR	Vac	2	1200	4	83.2	101.3	
	3794D	751	MR	Vac	2	1200	3	81.9	97.9	
	3794E	751	MR	Vac	2	1200	5	83.3	101.5	
	3794E	751	MR	Vac	2	1200	3	84.9	101.6	67
	3792	821	MR	Vac	2	1200	3	84.9	97.0	58
	3792	821	MR	Vac	2	1200	1	83.2	98.5	54

TABLE V (Cont.)

ROOM TEMPERATURE TENSILE PROPERTIES AND OXYGEN VALUES

Test	Bar	Alloy	Sample Location	Atm.	Anneal Hours	°C	Elongation (%)	Yield (ksi)	UTS (ksi)	O_2 (ppm)
45	2791B	751	MR				4	83.1	101.0	46
	2791B	751	MR				3	82.5	101.0	
	2791C	751	MR				4	83.3	103.1	
	2791C	751	MR				3	80.7	95.9	
	2791D	751	MR				4	84.2	104.3	47
	2791D	751	MR				4	81.9	99.3	
	3793	750	MR				1	80.0	94.1	
	3793	750	MR				3	82.9	99.4	
46	4791AA	751	MR	Vac	2	1200	7	83.1	109.8	
	4791AA	751	MR	Vac	2	1000	9	84.1	118.8	
47	4791BB	751	MR	Vac	2	1200	7	84.0	112.9	
	4791BB	751	MR	Vac	2	1000	10	83.3	118.2	
	4792	751	MR	Vac	2	1200	9	84.7	118.8	
	4792	751	MR	Vac	2	1000	5	83.3	105.4	
48	4791A*	751	MR	Vac	2	1200	10	84.0	119.9	14
	4791A	751	MR	Vac	2	1000	6	81.0	109.7	15
	4792B	751	MR	Vac	2	1200	3	80.8	102.1	25
	4792B	751	MR	Vac	2	1000	5	82.5	108.4	22
	4791A	751	MR	Vac	2	1200	9	82.3	116.2	18
	4791A	751	MR	Vac	2	1200	8	81.9	109.8	21
49	4791B	751	C	Vac	2	1200	4	76.4	91.3	38
	4791C	751	C	Vac	2	1200	4	78.2	95.7	26
	4791S	751	C	Vac	2	1200	10	83.3	119.2	15
	4791L	751	C	Vac	2	1200	7	76.4	91.3	

TABLE V (Cont.)

ROOM TEMPERATURE TENSILE PROPERTIES AND OXYGEN VALUES

Test	Bar	Alloy	Sample Location	Atm.	Anneal Hours	°C	Elongation (%)	Yield (ksi)	UTS (ksi)	0_2 (ppm)
50	6791A	901	MR	Vac	2	1200	10	N/A	N/A	47
	6791A	901	MR	Vac	2	1200	6	N/A	N/A	46
	6792A	821	MR	Vac	2	1200	12	86.7	127.5	30
	6792A	821	MR	Vac	2	1200	6	83.3	107.1	26
	6793A	750	MR	Vac	2	1200	5	N/A	N/A	
	6793A	750	MR	Vac	2	1200	7	N/A	N/A	
	6794-1	751	C	Vac	2	1200	9	85.8	118.0	
	6794-2	751	MR	Vac	2	1200	8	85.9	118.0	16
51	6794-2	751	MR	Vac	2	1200	9	86.6	123.0	14
	6794-3	751	C	Vac	2	1200	7	84.8	114.0	17
	8791AL	751	MR	Vac	2	1200	10	83.3	116.0	
52	8791AL	751	MR	Vac	2	1200	8	83.1	114.0	
	8791BL	751	MR	Vac	2	1200	8	81.2	116.0	
	8791BL	751	MR	Vac	2	1200	8	82.2	111.0	
	8791A	751	MR	Vac	2	1200	11	84.9	123.0	13
53	8791A	751	MR	Vac	2	1200	10	83.5	120.0	14
	8791B	751	MR	Vac	2	1200	8	83.9	120.0	14
	8791B	751	MR	Vac	2	1200	8	85.3	121.0	13
	8791C	751	C	Vac	2	1200	0	72.0	78.0	113
54	8791D	751	MR	Vac	2	1200	6	87.3	117.0	24
	8791D	751	MR	Vac	2	1200	5	84.5	104.0	29
	8791E	751	MR	Vac	2	1200	6	82.1	110.0	
55	8791E	751	MR	Vac	2	1200	8	83.2	116.0	
	8791F	751	C	Vac	2	1200	5	80.2	101.0	
	8791H	751	MR	Vac	2	1200	6	85.2	115.0	15

TABLE V (Cont.)

ROOM TEMPERATURE TENSILE PROPERTIES AND OXYGEN VALUES

Test	Bar	Alloy	Sample Location	Atm.	Anneal Hours	°C	Elongation (%)	Yield (ksi)	UTS (ksi)	O_2 (ppm)
56	8791H	751	MR	Vac	2	1200	7	86.2	123.0	15
	8791G	751	C	Vac	2	1200	5	84.2	112.0	15
	12791A	751	MR	N_2	2	1200	11	86.6	126.0	
57	12791	751	MR	N_2*	2	1200	6	86.6	121.0	
	12791B	751	MR	N_2	2	1200	6	84.9	111.0	
	12791B	751	MR	N_2	2	1200	10	86.8	123.0	
	12791C	751	MR	Vac	2	1200	8	87.2	119.0	
58	12791C	751	MR	Vac	2	1200	7	84.8	117.0	
	12791D	751	MR	Vac	2	1200	7	84.8	113.0	
	12791D	751	MR	Vac	2	1200	5	84.9	112.0	
	12791E	751	MR	N_2	2	1200	10	85.8	119.0	
	12791E	751	MR	Vac	2	1200	10	84.8	118.0	
59	12791F	751	MR	Vac	2	1200	10	86.0	122.0	
	12791F	751	MR	N_2*	2	1200	10	85.6	121.0	
	12791G	751	MR	N_2*	2	1200	7	84.2	113.0	
	12791G	751	MR	N_2	2	1200	7	84.9	117.0	
	12791H	751	MR	Vac	2	1200	7	84.2	115.0	15
60	12791H	751	MR	Vac	2	1200	7	85.2	121.0	16
	12791K	751	MR	Vac	2	1200	5	86.3	116.0	16
	12791K	751	MR	Vac	2	1200	7	85.5	117.0	
	2802A	751	MR	Vac	2	1200	7	84.9	119.0	16
61	2802A	751	MR	Vac	2	1200	11	85.3	121 0	16
	12791M	751	MR	Vac	2	1200	11	84.9	123.0	17
	12791M	751	MR	Vac	2	1200	10	83.5	117.0	14
	2802D	751	MR	Vac	2	1200	10	83.1	120.0	16

TABLE V (Cont.)

ROOM TEMPERATURE TENSILE PROPERTIES AND OXYGEN VALUES

Test	Bar	Alloy	Sample Location	Atm.	Anneal Hours	°C	Elongation (%)	Yield (ksi)	UTS (ksi)	O_2 (ppm)
62	2802D	751	MR	Vac	2	1200	9	83.5	123.0	
	2802B	751	MR	Vac	2	1200	9	85.5	126.0	15
	2802B	751	MR	Vac	2	1200	9	84.6	121.0	
	1801A	901	MR	Vac	2	1200	17	88.5	137.0	4,6
63	1801A	901	MR	Vac	2	1200	16	89.1	137.0	2,3
64	2803B	751	MR	Vac	2	1200	8	84.5	117.0	13
	2803B	751	MR	Vac	2	1200	9	84.2	121.0	13
65	2803D	751	MR	Vac	2	1200	6	83.9	119.0	19
	2803D	751	MR	Vac	2	1200	6	81.1	104.0	17
66	2803F	751	MR	Vac	2	1200	6	85.8	121.0	19
	2803F	751	MR	Vac	2	1200	4	84.6	109.0	22

Center-to-edge density gradients were determined by taking about a 0.1" slice of the bar and sectioning it as sketched:

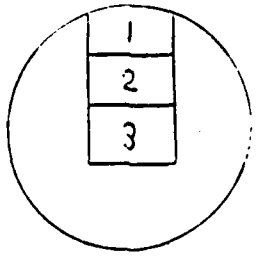


Table VI contains the density determinations made for Tests 5 to 42. These densities are for the whole bars. Table VII contains the density data for Runs 43 to 66. In a few cases, the density was determined from a large piece cut from the center of the bar.

TABLE VI

SINTERED DENSITIES - TESTS 5 TO 42

Test	Lot	Alloy	Pressing Pressure (ksi)	Sintered Size (Dia. x Length)	Sintering Furnace	Sintered Density (%)	Comments
5	3781	751	35	0.64 x 1		94.8	MD-151 Cu powder
	3782	751	35	0.64 x 1	Brew	94.9	MD-151 (-325 mesh)
	4781	WN-107	35	0.64 x 1		99.6	
6	3781	751	35	0.64 x 1	Brew	95.2	
	3782	751	35	0.64 x 1		95.3	
7	3781	751	35	0.64 x 1		95.0	
	3782	751	35	0.64 x 1	Brew	95.3	
	4782	751	35	0.64 x 1		95.2	MD-301 Cu powder
	3781	751	35	0.64 x 1		94.9	All presintered in wet H_2 at 1200 °C
8	3782	751	35	0.64 x 1	Brew	95.1	
	4782	751	35	0.64 x 1		94.9	
9	3782	751	35	0.64 x 1		96.1	Slower ramp rates used
	4781	WN-107	35	0.64 x 1	Brew	98.2	between 1000 to 1650 °C
	4782	751	35	0.64 x 1		96.1	
	4781	WN-107	35	0.64 x 1		99.3	
	4782	751	20	0.64 x 1		94.7	
10	5781	901	35	0.64 x 1	Brew	98.4	
	5782	821	35	0.64 x 1		98.5	
	5783	750	35	0.64 x 1		95.2	
	4781	WN-107	35	0.64 x 1		99.4	Sintering temperature
	4782	751	20	0.64 x 1		94.9	lowered 50 °C for this test

TABLE VI (Cont.)

SINTERED DENSITIES - TESTS 5 TO 42

Test	Lot	Alloy	Pressing Pressure (ksi)	Sintered Size (Dia. x Length)	Sintering Furnace	Sintered Density (%)	Comments
11	5781	901	35	0.64 x 1	Brew	98.8	
	5782	821	35	0.64 x 1		98.7	
	5783	750	35	0.64 x 1		95.4	
	4781	WN-107	35	0.64 x 1		99.0	Used 630 °C hold in-
	4782	751	35	0.64 x 1		96.0	stead of 1000 °C
12	5781	901	35	0.64 x 1	Brew	98.5	
	5782	821	35	0.64 x 1		98.7	
	5783	750	35	0.64 x 1		95.5	
13	4781	WN-107	35	0.64 x 1		99.2	
	4782	751	35	0.64 x 1		96.1	Cooling rate lowered
	5781	901	35	0.64 x 1	Brew	98.4	
	5782	821	35	0.64 x 1		98.8	
	5783	750	35	0.64 x 1		95.7	
	4781	WN-107	35	0.64 x 1		99.5	Used faster ramp be-
	4782	751	35	0.64 x 1		95.4	tween 1000 to 1600 °C
14	5781	901	35	0.64 x 1	Brew	99.1	
	5782	821	35	0.64 x 1		98.1	
	5783	750	35	0.64 x 1		95.1	
	4782	751	35	0.64 x 1	Brew	93.7	Blend Attritor milled
15	5783	750	35	0.64 x 1	Brew	96.2	Blend Attritor milled
	6781	751	35	0.64 x 1	Brew	97.9	Ni-Cu-Co co-reduced
	4781	WN-107	35	0.64 x 1		99.7	Sintered in H_2 and
	4782	751	35	0.64 x 1	V.I.	94.9	vacuum

TABLE VI (Cont.)

SINTERED DENSITIES - TESTS 5 TO 42

Test	Lot	Alloy	Pressing Pressure (ksi)	Sintered Size (Dia. x Length)	Sintering Furnace	Sintered Density (%)	Comments
16	5781	901	35	0.64 x 1		99.3	
	5782	821	35	0.64 x 1		97.6	
	5783	750	35	0.64 x 1		94.7	
	4781	WN-107	35	0.64 x 1	#3 Muffle	99.9	
	4782	751	35	0.64 x 1		94.9	
	5781	901	35	0.64 x 1		99.7	
17	5782	821	35	0.64 x 1		98.1	
	5783	750	35	0.64 x 1		94.7	
	6781	751	35	0.64 x 1		97.0	
	4781	WN-107	35	0.64 x 1	#3 Muffle	99.9	Slower stoke than
	4782	751	35	0.64 x 1		92.9	Test 17
18	5781	901	35	0.64 x 1		98.8	
	5782	821	35	0.64 x 1		96.2	
	5783	750	35	0.64 x 1		92.2	
	6781	751	35	0.64 x 1		94.8	
	4781	WN-107	35	0.64 x 1		99.8	
	4782	751	35	0.64 x 1		94.0	
	5781	901	35	0.64 x 1	#3 Muffle	99.5	
19	5782	821	35	0.64 x 1		99.7	
	5783	750	35	0.64 x 1		93.6	
	6781	751	35	0.64 x 1		96.0	
	6782	751	35	0.64 x 1		93.7	Very fine Cu powder
	6783	751	35	0.64 x 1		99.0	M-30 tungsten powder
	4781	WN-107	35	0.64 x 1		99.7	

TABLE VI (Cont.)

SINTERED DENSITIES - TESTS 5 TO 42

Test	Lot	Alloy	Pressing Pressure (ksi)	Sintered Size (Dia. x Length)	Sintering Furnace	Sintered Density (%)	Comments
20	4782	751	35	0.64 x 1	Brew	94.4	
	6782	751	35	0.64 x 1		94.3	
	6783	751	35	0.64 x 1		C	(M-30 tungsten powder)
	4781	WN-107	35	0.64 x 1	Small Induction	98.4	
21	4782	751	35	0.64 x 1		97.9	
	6782	751	35	0.64 x 1		95.0	
	6783	751	35	0.64 x 1		C	
	4781	WN-107	35	0.64 x 1	Large Stoke	99.4	
22	4782	751	35	0.64 x 1		98.0	
	6782	751	35	0.64 x 1		97.8	
	6783	751	35	0.64 x 1		C	
	4781	WN-107	35	0.64 x 1		98.3	3 to 5 minutes at high point
23	4782	751	35	0.64 x 1	V.I.	91.9	
	8781	751	35	0.64 x 1		93.3	
	4781	WN-107	35	0.64 x 1	Brew	99.3	
24	4782	751	35	0.64 x 1		94.5	
	8781	751	35	0.64 x 1		C	
25	4781	WN-107	35	0.64 x 1		99.4	Used lower temperature
	4782	751	35	0.64 x 1	Brew	93.4	
	8781	751	35	0.64 x 1		C	

TABLE VI (Cont.)

SINTERED DENSITIES - TESTS 5 TO 42

Test	Lot	Alloy	Pressing Pressure (ksi)	Sintered Size (Dia. x Length)	Sintering Furnace	Sintered Density (%)	Comments
26	4781	WN-107	35	0.64 x 1		99.5	
	4782	751	35	0.64 x 1	Brew	93.7	Used even lower
	8781	751	35	0.64 x 1		C	temperature
	4781	WN-107	35	0.64 x 1		99.6	
27	4782	751	35	0.64 x 1	Brew	94.5	Slow ramp 1400 to 1500
	8781	751	35	0.64 x 1		C	°C
	4781	WN-107	35	0.64 x 1		99.7	
28	4782	751	35	0.64 x 1	Brew	99.3	
	8781	751	35	0.64 x 1		C	
	4781	WN-107	35	0.64 x 1	#3 Muffle	99.7	
29	4782	751	35	0.64 x 1		94.2	
	8781	751	35	0.64 x 1		C	
	6783	751	35	0.64 x 1		98.6	
	4781	WN-107	35	0.64 x 1		80.4	Run terminated after 1400 °C hold
30	4782	751	35	0.64 x 1	Brew	81.1	
	8781	751	35	0.64 x 1		94.5	
	4781	WN-107	From Test 30			99.6	
	4782	751				94.0	
	8781	751				C	
31	4781	WN-107	35	0.64 x 1	Brew	99.7	
	4782	751	35	0.64 x 1		93.7	
	8781	751	30	0.64 x 1		C	
	8781	751	30	1.09 x 3		99.1	
	4781	WN-107	35	0.64 x 1		99.6	

TABLE VI (Cont.)

SINTERED DENSITIES - TESTS 5 TO 42

Test	Lot	Alloy	Pressing Pressure (ksi)	Sintered Size (Dia. x Length)	Sintering Furnace	Sintered Density (%)	Comments
32	4782	751	35	0.64 x 1	V.I.	94.4	
	8781	WN-107	30	0.64 x 1		C	
	4781	WN-107	35	0.64 x 1		99.8	
	8781	751	30	0.64 x 1	Brew	C	
	9781	WN-107	30	0.64 x 1		99.1	
33	9782	751	30	0.64 x 1		98.3	
	8781	751	30	1.09 x 3		99.7	
	8781	751	30	1.09 x 3		99.3	
	9782	751	30	1.09 x 3		98.9	
	4781	WN-107	35	0.64 x 1		99.9	
	4782	751	35	0.64 x 1		94.1	Sintered in N_2
34	8781	751	30	0.64 x 1	Brew	99.1	
	9781	WN-107	30	0.64 x 1		99.8	
	9782	751	30	0.64 x 1		98.3	
35	8781	751	30	0.64 x 1	Brew	C	
	10781	751	24	1.10 x 3		98.9	
	8781	751	30	0.64 x 1		C	
36	4781	WN-107	35	0.64 x 1	Brew	99.8	
	10781	751	24	1.10 x 3		98.4	
	10781	751	24	1.10 x 3	Brew	97.8	
37	10781	751	30	1.10 x 3		98.9	
	10781	751	24	1.10 x 3		98.8	

TABLE VI (Cont.)

SINTERED DENSITIES - TESTS 5 TO 42

Test	Lot	Alloy	Pressing Pressure (ksi)	Sintered Size (Dia. x Length)	Sintering Furnace	Sintered Density (%)	Comments
38	10781	751	30	1.10 x 3	V.I.	99.3	
	10782	751	27	1.35 x 4		98.9	
	4781	WN-107	30	0.64 x 1		99.8	
39	8781	751	30	0.64 x 1	Brew	C	
	10782	751	30	1.35 x 6		98.4	
	10782	751	35	1.35 x 6		98.6	
40	10782	751	30	1.35 x 6	Brew	99.3	
41	10782	751	30	1.35 x 6	Brew	98.8	
	1791	751	30	1.35 x 6		99.5	
	4781	WN-107	30	0.64 x 1		99.8	
42	9781	WN-107	30	0.64 x 1	Brew	99.8	
	2791	751	30	0.64 x 1		99.6	
	2791	751	30	1.35 x 6		99.6	

TABLE VII

SINTERED DENSITIES - TESTS 43 TO 66

Test	Alloy	Bar	Sintered Density (%) Sliced Sections 1	2	3	Center Section
	1	2791A	99.63	98.87	98.48	
43	751	3794A	99.64	98.75	98.46	
	751	3794B	99.46	99.07	98.78	
	901	3791	99.46	99.39	99.38	
	821	3792	99.41	98.76	98.97	
44	751	3794C	99.37	98.72	98.61	
	751	3794D	99.48	98.57	98.71	
	751	3794E	99.73	98.32	98.83	
	750	3793	99.74	98.99	98.90	
	751	2791B	99.55	98.93	98.85	
45	751	2791C	99.78	98.84	99.46	
	751	2791D	99.84	98.75	98.58	
46	751	4791AA	99.41	99.28	99.24	99.20
47	751	4791BB	99.60	99.59	99.24	99.49
	751	4792	99.70	99.42	99.19	99.63
48	751	4791CC	99.58	99.43	99.33	99.38
	751	4792B	99.75	99.71	99.68	99.74
	751	4791A				99.19
	751	4791B				99.33
49	751	4791C				99.22
	751	4791S				99.72
	751	4791L				99.38
51	751	6794-3	99.72	99.43	99.29	
52	751	L8791A	99.73	99.53	99.41	
	751	L8791B	99.63	99.46	99.34	
53	751	8791A	99.84	99.71	99.38	
	751	8791B	99.79	99.60	99.46	
54	751	8791C	99.98	99.48	98.93	
55	751	8791E	99.79	99.42	99.29	
	751	8791F	99.72	99.58	99.44	

TABLE VII (Cont.)

SINTERED DENSITIES - TESTS 43 TO 66

Test	Alloy	Bar	Sintered Density (%) Sliced Sections 1	2	3	Center Section
56	751	8791G	99.82	99.64	99.50	
	751	8791H	99.87	99.71	99.65	
57	751	12791A	99.74	99.51	99.28	
	751	12791B	99.80	99.57	99.20	
58	751	12791C	99.61	99.46	99.38	
	751	12791D	99.77	99.38	99.12	
	751	12791E	99.77	99.47	99.20	
59	751	12791F	99.78	99.39	99.26	
	751	12791G	99.79	99.40	99.08	
60	751	12791H	99.74	99.41	99.08	
	751	12791K	99.78	99.58	99.11	
61	751	12791M	99.84	99.76	99.72	
	751	2802A	99.88	99.68	99.51	
62	751	2802B	99.78	99.56	99.50	
	751	2802D	99.77	99.61	99.37	
63	901	1801A			99.39	
64	751	2803B	99.93	99.79	99.57	
65	751	2803D	99.94	99.90	99.52	
66	751	2803F	99.98	99.21	98.71	

EXPERIMENTS AND RESULTS

Our experimental work can be divided into two general areas which encompass the three phases of the contract. The first was our efforts to develop good density in the 751 alloy. Once the density was achieved we directed our efforts to improving the tensile properties and uniformity of the large bars (1.35" green diameter) of the 751 alloy.

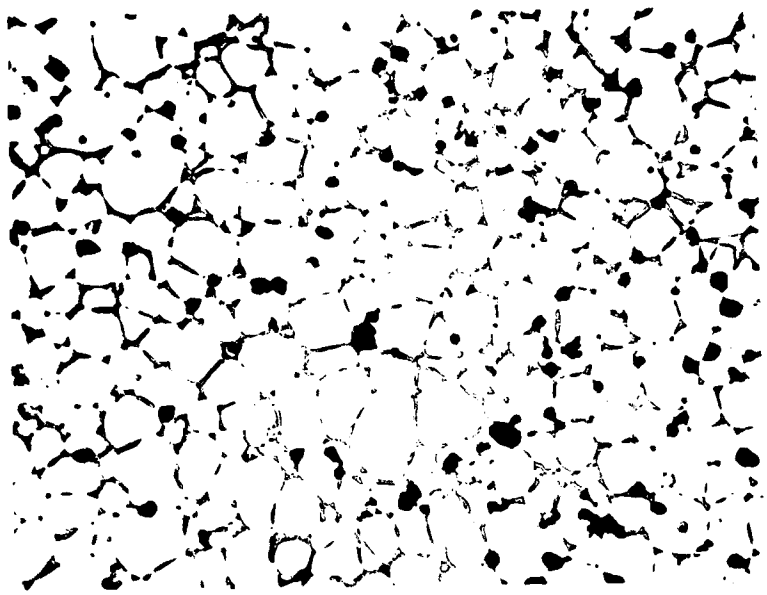
A. Development of High Density

In our initial work we used small diameter bars (0.64" green) to develop sintering schedules. Blends were made using a Sylvania M-55 tungsten powder with a FSSS of 5.25. We chose M-55 tungsten powder based on our experience with tungsten-nickel-iron systems in which the best density is obtained using M-55 tungsten powder. Our first few blends used a MD-151 copper powder which had a FSSS of 14.5. From the appearance of the parts, we decided this was too coarse and switched to a MD-301 with a FSSS of 8.7 for the remainder of the contract. Also included in these initial tests were parts made of Sylvania's WN-107 alloy. The composition of the alloy is 97.3W-1.35Ni-1.35Fe.

1. Sintering Schedule Variations

In Tests 5 to 14, which were all run in the Brew furnace, we tried variations in the sintering schedule to develop good density in the 751 alloy. The things we varied were heating rates to hold points, the temperature and time of these holds, and the cooling rate from the sintering temperature. None of these variations proved successful as the 751 density ranged between 94.7% and 96.1%. However, densities for the WN-107 alloy ranged from 98.2% to 99.6%.

Figure 1 shows an interesting comparison in the microstructure of these two alloys. In addition to porosity, the 751 alloy has squarer grains with several places where four grains meet instead of three.



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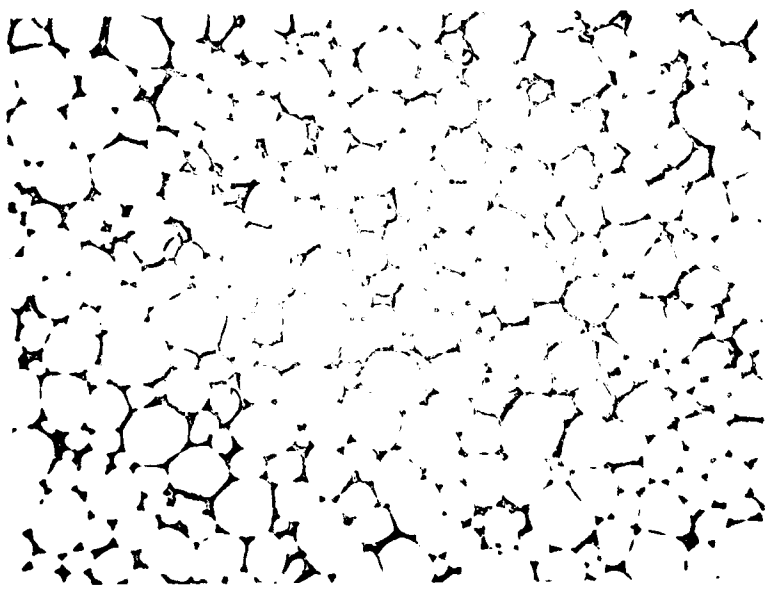


WN-107

Figure 1. Light Micrograph, 100X of 0.64" diameter x 1" long samples sintered in Test 10

2. Prealloyed Matrix and Attritor Milling

During sintering of a W-Ni-Fe alloy, the nickel will melt at about 1455 °C and the alloy formed will have roughly the same melting point. When copper is added to the system, the situation is more complex because the copper will melt at about 1080 °C and continue to form new matrix compositions dissolving more nickel, iron, and tungsten as the temperature increases. It is for this reason we tried to prealloy the matrix elements.

In Test 15 we did this by milling the blends in a small Attritor mill using tungsten carbide balls as a grinding medium. Only a small amount of powder was milled and the bars were difficult to process. We had mixed results with this test as one piece had a slightly lower density than normal and the other had a slightly higher density than normal. In our other attempt, the nickel, copper, and cobalt were coreduced from dried solutions of the salts. Iron was not included because of difficulties in reducing it along with the others. The coreduced powders were mixed with the tungsten and iron powder to produce the blend. We sintered samples of this blend in Tests 15, 17, 18, and 19. Densities ran about 2% higher on this blend compared to standard blends.

3. Alloy Variations

Starting with Test 10 we also began looking at variations of the 751 alloy. These included a 901, 821, and 750, all having 97.1% tungsten. The general result from these tests (10 to 19) was that density went up as the copper content decreased.

4. Tungsten Particle Size

In Test 19, which was sintered in a muffle furnace, we included a piece that had been pressed from a blend made with a finer tungsten powder (M-30). This piece achieved a density of 99.0% which

was the best we had seen in the 751 alloy. Subsequent tests in the Brew furnace with this blend yielded pieces that were cracked as shown in Figure 2. Because of the cracking, we couldn't determine density, but the microstructure (Figure 3) revealed a dense structure. We continued to get cracking in samples made with M-30 blends until we sintered a larger diameter bar (1.1") in Test 31. This bar had no cracking and a density of 99.1%. Although further tests showed reasonable density and no cracking, we did see a defect we called ring porosity. Figures 4 and 5 show this defect which is a narrow band of small pores under the surface of the bar.

Our next step was to increase the diameter of the bar to 1.35" which was the size we wanted to make for the contract. Densities for these bars ran between 98.4% and 99.3%.

In hopes of raising the density, we went to even a finer tungsten powder (M-17). These bars gave us better than 99.5% density, so we stayed with M-17 tungsten powder for the rest of the contract. One interesting result was that a small diameter bar (0.64) made from a M-17 blend sintered to a good density and didn't crack.

5. Pressing Pressure

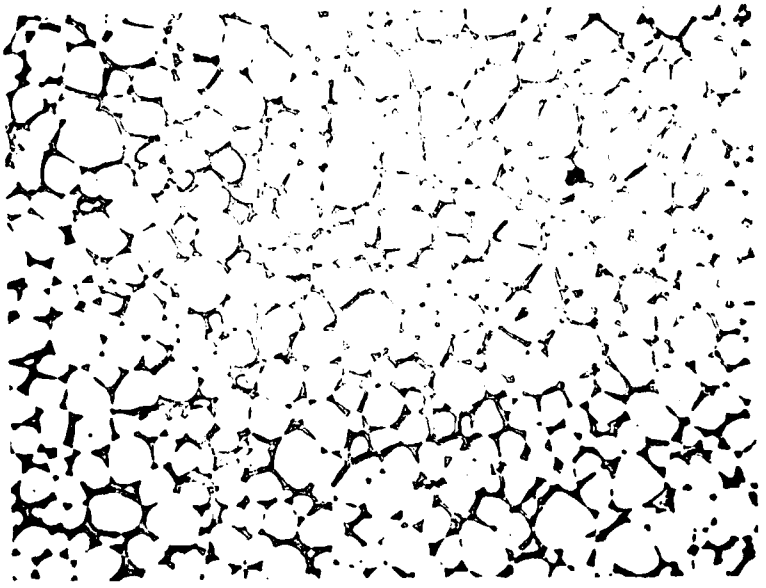
For the 1.1"-diameter bars we found in Tests 37 and 38 that a lower pressing pressure gave lower sintered densities. We didn't see this in the 1.35"-diameter bars.

6. Furnaces and Atmosphere

Test 22 was run through a large muffle furnace using a slow stoke and a wet atmosphere. This run gave us our best density (97.8%) for a 0.64"-diameter piece made with a M-55 blend. However, a piece from a M-30 blend cracked badly.

1.5X

Figure 2. Macrophotograph showing cracks in sintered 751 alloy sample made from M-30 tungsten powder



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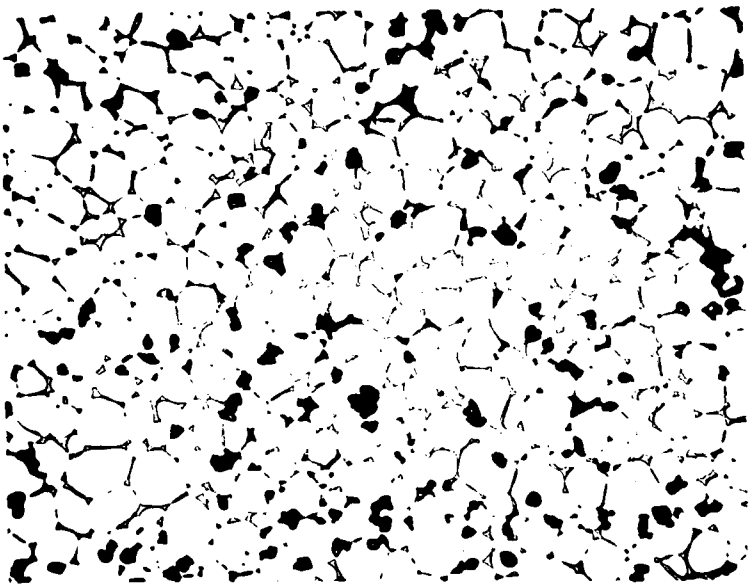
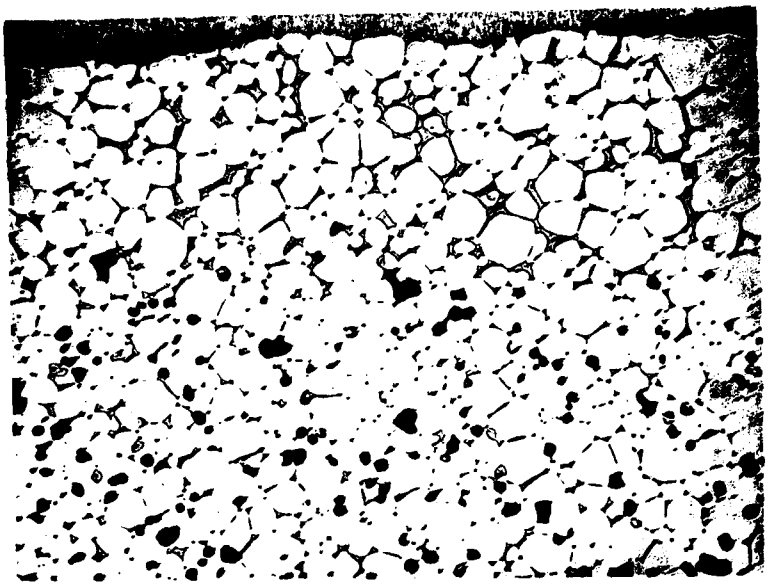


Edge

Figure 3. Light Micrograph, 100X of sample in Figure 2



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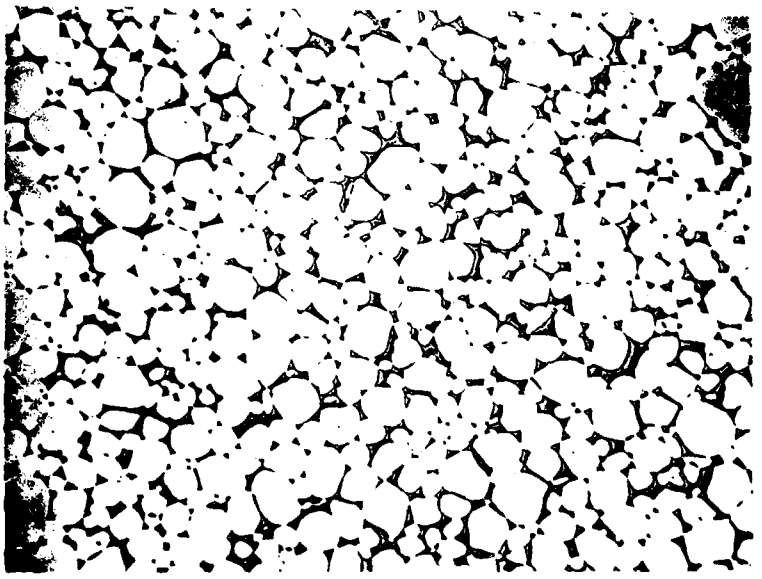


Center

Figure 4. Light Micrographs showing ring porosity in 75 alloys (1.1"-diameter bars) 100X

Top

Bottom

Figure 5. Macrophotograph of [illegible] porosity in 751 alloy (1.1"-diameter bar)

Other tests were run in a smaller muffle furnace in which dry hydrogen was used but probably had a dew point 0 °C. No density improvement was noted but a piece from a M-30 blend sintered without cracking where similar pieces sintered in the Brew cracked.

Another interesting test (34) was run in the Brew furnace using nitrogen after the hold at 1000 °C. No density improvement was noted for the 751 alloy but no cracking occurred in the piece made from a M-30 blend.

B. Improvement of Properties

Once we were able to achieve good densities in the 1.35"-diameter bars, we started a series of tests (43 to 45) to determine uniformity within and among sintering runs. Also included in these tests were bars of the alloy variations we had tried in the small pieces. Although the bars appeared to be uniform, they had very poor elongations ranging from 1% to 5%. Our next series of tests was then aimed at improving the tensile properties.

1. Heating Rates

In Tests 46 and 47 we cut the ramp rates (heating rates) to one-third of what we had been using for the first and second ramp. This gave us an immediate improvement with elongations of 5% to 10%. A further reduction was tried in Test 48 for the second ramp with no apparent improvement.

In Test 49 we lowered the third ramp rate and saw no improvement in properties. The interesting result of this test was that tensile bars taken from the center of the large bars had elongations of only 4%. Bars taken from the bars side-by-side, as we had been doing, had elongations of 8% and 9%. Tests 57 and 59 were run with a fast first ramp to show the critical ramp was the second. Test 52 was run at a 25 °C higher sintering temperature, and Test 55 was run 25 °C lower.

2. Cooling Rates

In Test 51 we lowered the cooling rate from the sintering temperature from 30 mv per hour to 1 mv per hour. As a result, tensile samples taken from the center of the bars had comparable elongations to those taken near the edge. No improvement of the density gradient was noted. Most of the subsequent sintering runs were done with this slow cooling.

3. Atmosphere

In Tests 58, 64, 65, and 66 we added water to the hydrogen to vary the dew point from -30 °C to +20 °C. A normal dry run was about -40 °C. These tests showed a slight increase in oxygen in the bars with a corresponding decrease in tensile properties. We also developed porosity in the center of the bar sintered at +20 °C (Figure 6).

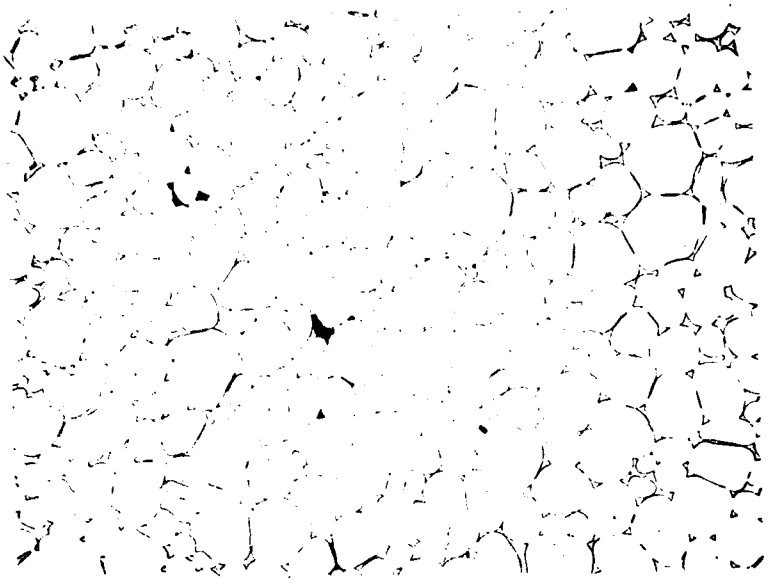
In Test 56 we doubled the hydrogen flow rate. No effect on properties was noted.

4. Alloy Variations

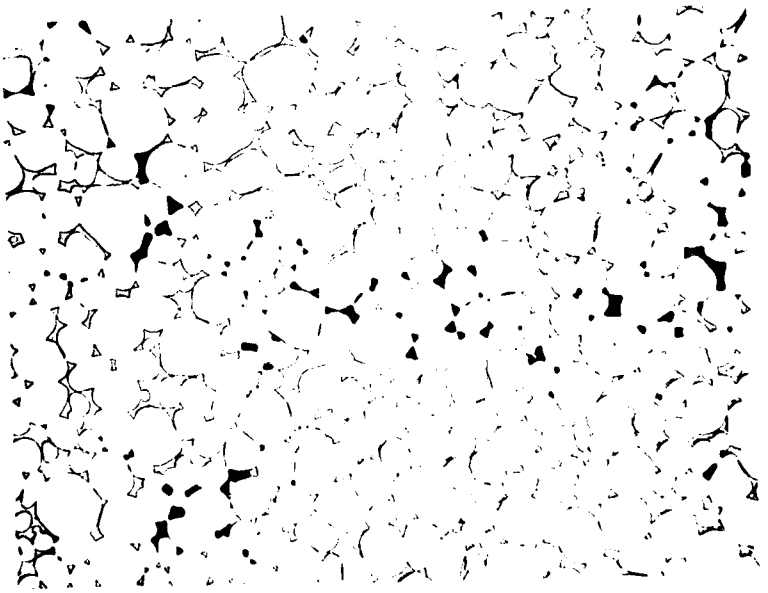
In Test 50 we looked at some of the same alloys we did in Tests 42 to 44. Like the 751 alloy, we showed a marked improvement in the tensile properties. We felt the tensile properties of the 901 alloy (no copper) should have been better so in Test 63 we sintered a bar of the 901 alloy made from M-55 powder instead of M-17. This test gave us elongations of 16% and 17% compared to 6% and 10% for the test made from a M-17 blend.

C. Uniformity of Properties

We looked at uniformity of properties in the 751 alloy in Test 43 to 45 and 64 to 66. We looked at only tensile properties, oxygen, and density in Tests 64 to 66.



Edge



Center

Figure 6. Light Micrograph showing porosity in 751 bar sintered at +20 °C dew point

1. Hardness

Table VIII contains the hardness values taken on bars sintered in Runs 43 to 45. They represent hardness taken across the face about 1" in from the end of the bar.

2. Composition

Table IX contains the composition determined by atomic absorption for bars in Runs 43 to 45. Samples were taken from a slice 1" in from the end of the bar.

3. Ultrasonic Inspection

All 12 bars from Tests 43 to 45 were ultrasonically scanned from both ends with no defects found. We used a 5 MHZ - 0.064"-diameter crystal.

4. Density and Tensile Properties

These results, as previously noted, are contained in Tables V and VII. Figures 7 to 12 are SEM photographs of the fracture surfaces from Tests 43 to 45.

5. Microstructure

Light micrographs from Tests 43 to 45 are shown in Figures 13 to 15.

TABLE VIII

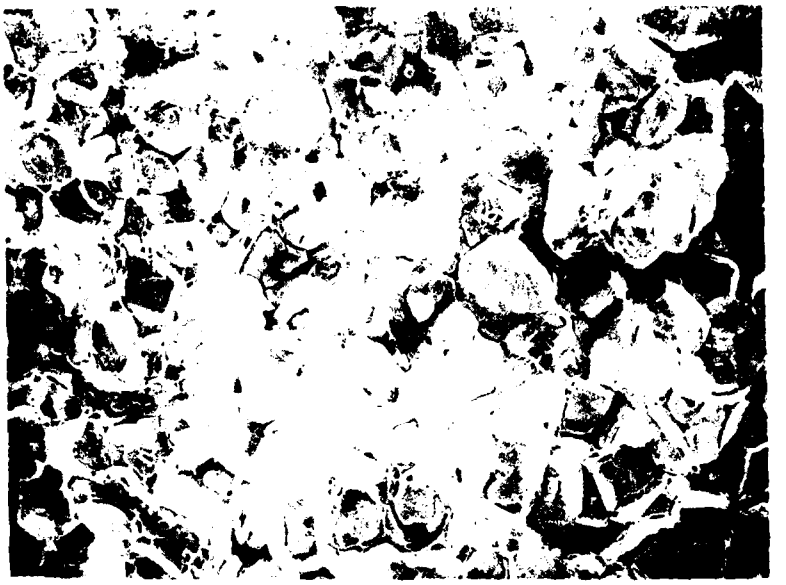
HARDNESS (R_C) - RUNS 43 TO 45

Lot	Run	Alloy	Hardness (Rockwell C) 1	2	3	4	5	6	7	Mean	S. Dev.
2791A	43	751	27.2	26.0	24.7	24.5	24.3	26.0	25.8	25.5	1.05
3794A	43	751	28.6	27.8	27.4	27.9	27.3	27.8	28.2	27.9	0.45
3794B	43	751	28.5	28.1	27.9	27.4	28.2	28.7	29.0	28.3	0.51
3791	43	901	27.3	27.7	27.4	27.3	27.9	27.5	26.5	27.4	0.44
3794C	44	751	28.1	28.1	27.8	27.2	27.7	28.0	28.8	28.0	0.49
3794D	44	751	28.7	28.3	27.5	27.2	27.2	28.9	29.8	28.2	0.98
3794E	44	751	29.3	28.2	27.8	27.9	27.5	28.3	28.3	28.2	0.57
3792	44	821	29.0	28.2	28.8	28.1	28.2	28.2	28.5	28.4	0.35
2791B	45	751	28.8	29.2	29.0	28.9	28.3	28.9	28.8	28.8	0.28
2791C	45	751	29.0	27.8	26.6	27.7	27.1	27.3	28.2	27.7	0.78
2791D	45	751	28.3	27.8	26.9	27.5	27.3	29.0	29.5	28.0	0.94
3793	45	750	27.9	26.5	27.2	26.8	27.2	28.3	30.2	27.7	1.25

TABLE IX

COMPOSITION - RUNS 43 TO 45

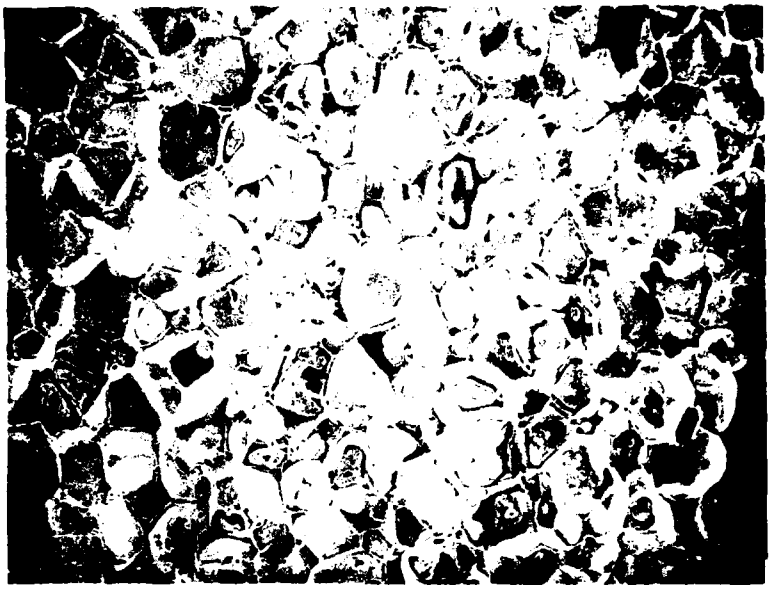
Lot	Run	Alloy	Composition, % (4 to 5)			
			Ni	Fe	Cu	Co
2791A	43	751	1.68	0.73	0.45	0.11
3794A	43	751	1.64	0.73	0.45	0.10
3794B	43	751	1.60	0.72	0.50	0.10
3791	43	901	1.89	0.90	<0.05	0.10
3794C	44	751	1.65	0.73	0.49	0.10
3794D	44	751	1.66	0.73	0.49	0.11
3794E	44	751	1.65	0.73	0.50	0.11
3792	44	821	1.83	0.82	0.19	0.10
2791B	45	751	1.64	0.72	0.48	0.10
2791C	45	751	1.59	0.72	0.50	0.11
2791D	45	751	1.60	0.71	0.49	0.10
3793	45	750	1.74	0.71	0.51	<0.01



200X

500X

Figure 7. SEM of fracture surface Test 43 - Bar 3794A



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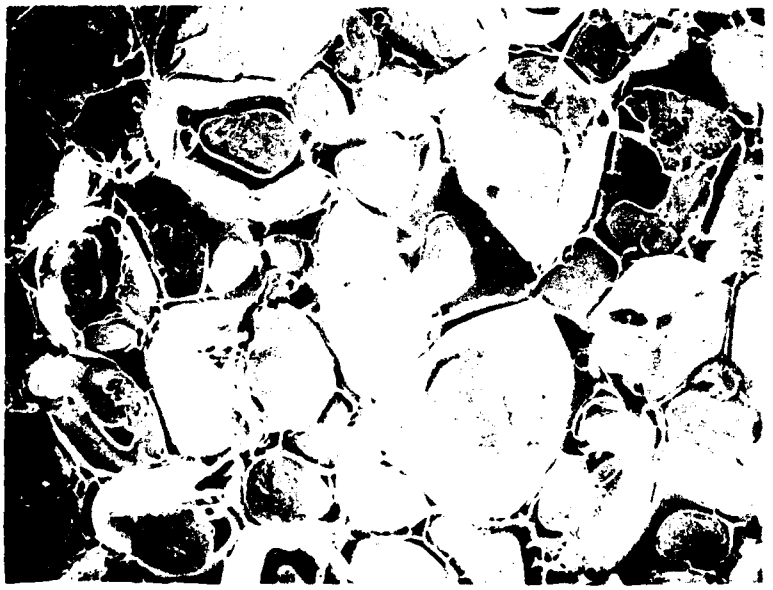

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Figure 8. SEM of fracture surface Test 43 - Bar 3794B

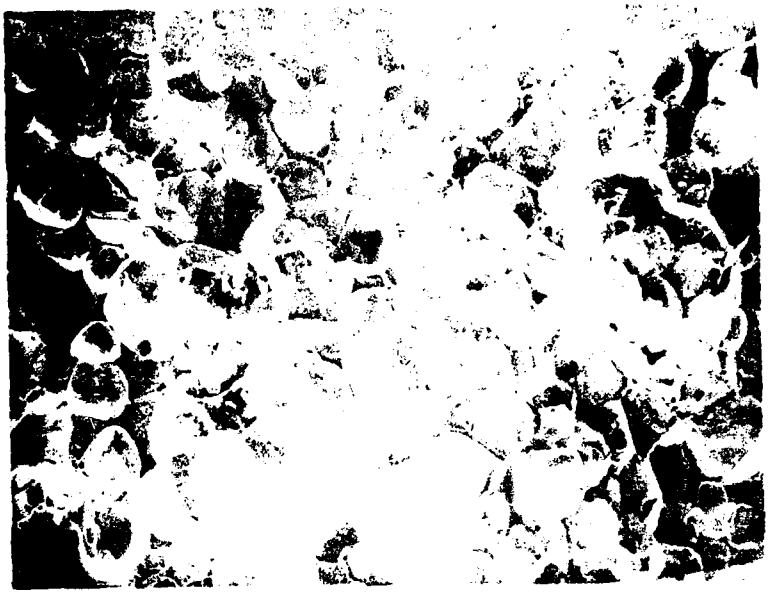

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Figure 9. SEM of fracture surface Test 44 - Bar 3794D

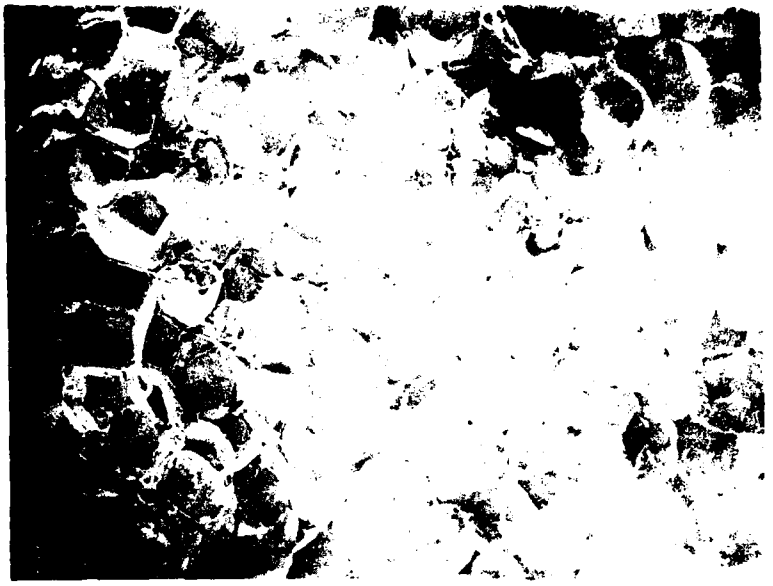

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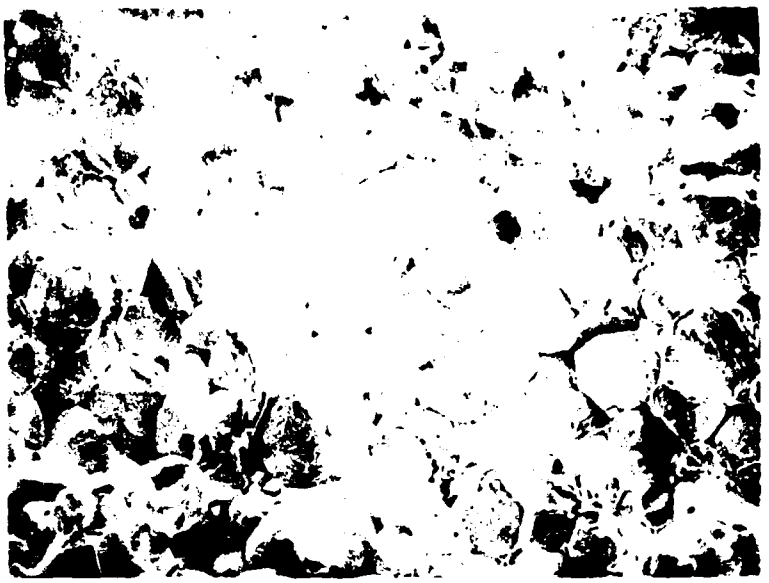
Figure 10. SEM of fracture surface Test 41 - Bar 3041



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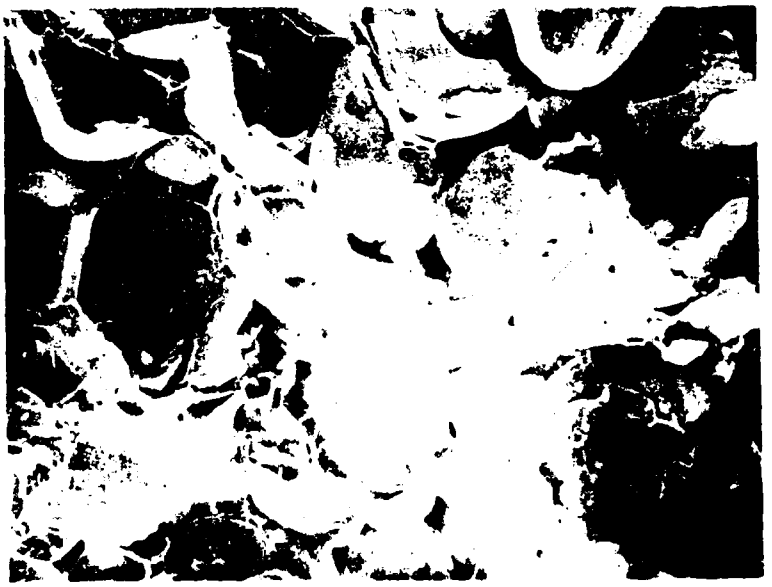
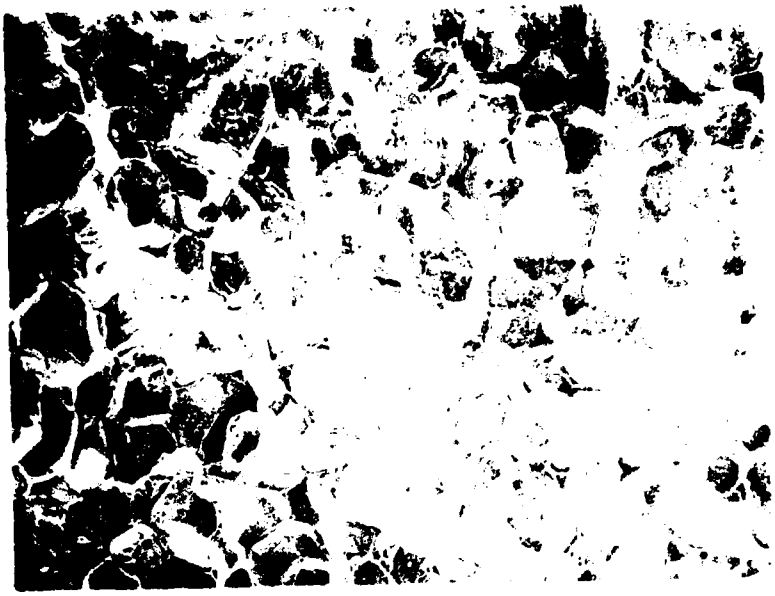


500X

Figure 11. SEM of fracture surface Test 45 - Bar 2791B



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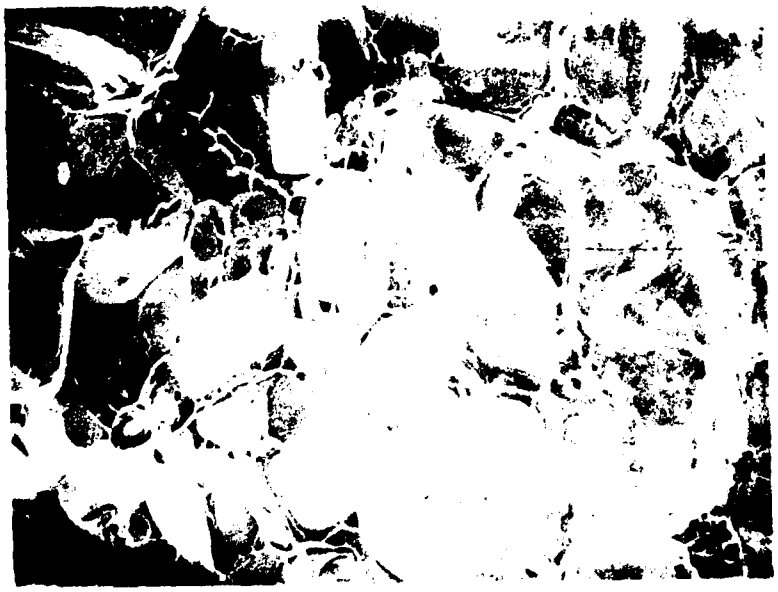
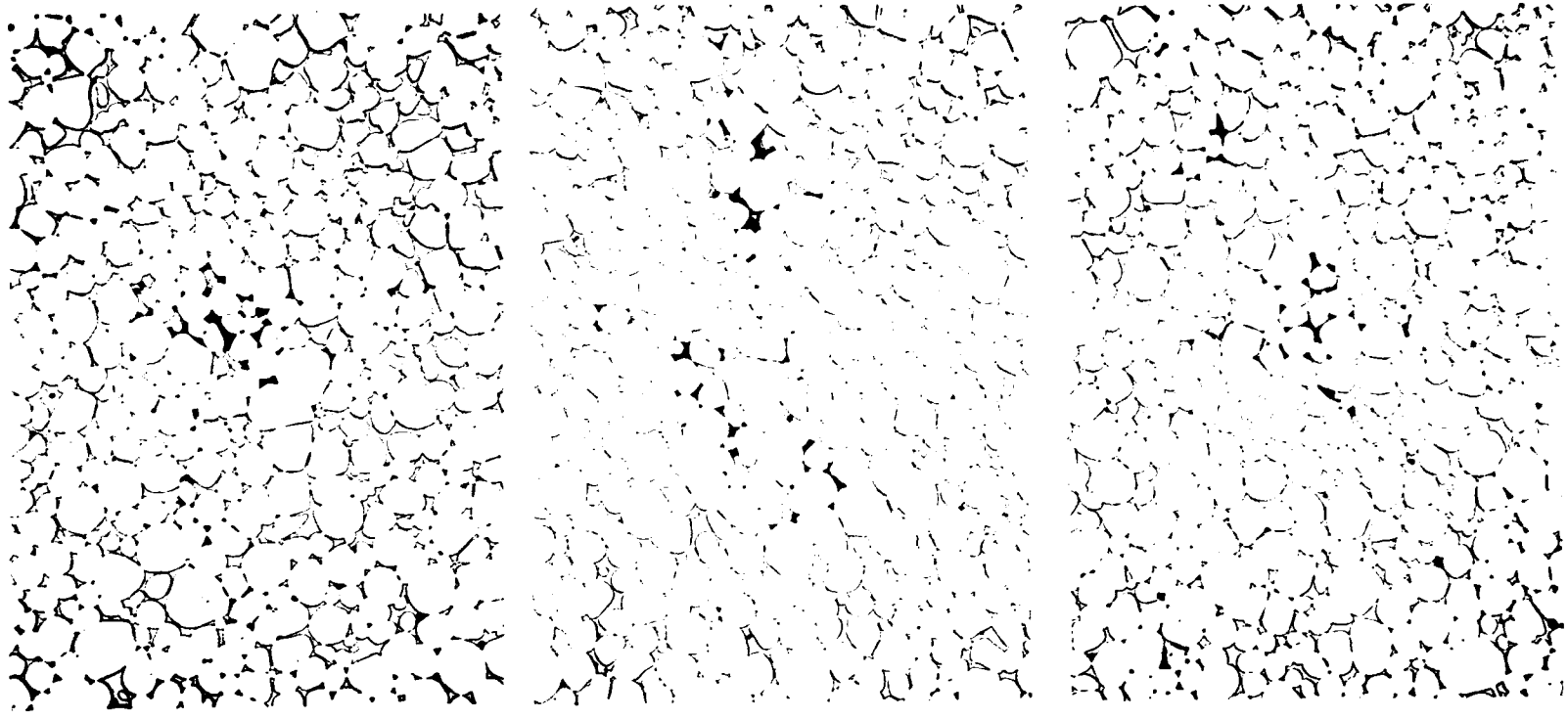


500X

Figure 12. SEM of fracture surface Test 15 Bar



2971A 3794B 3794A

Figure 13. Light Micrographs, 100X, from Test 43

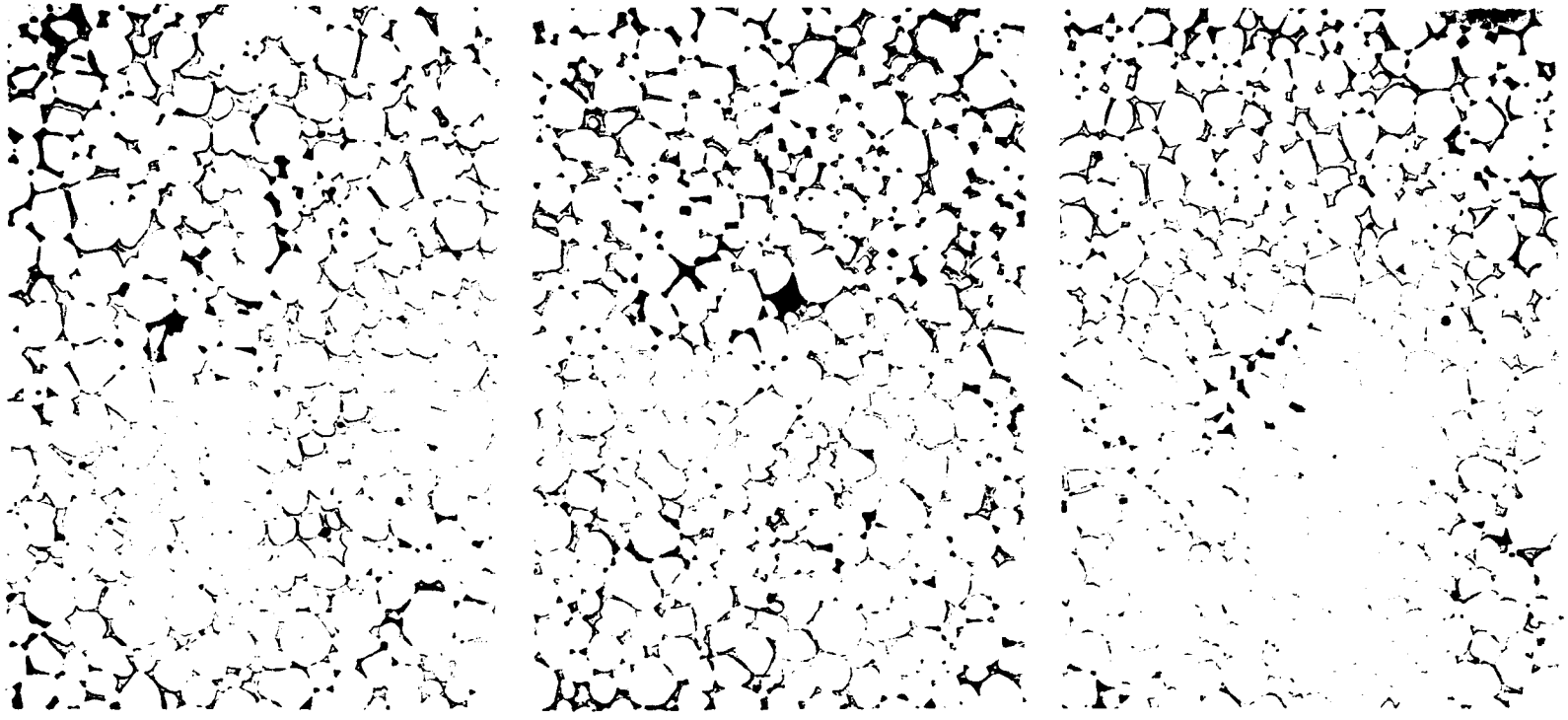


Figure 14. Light Micrographs, 100X, from Test 44

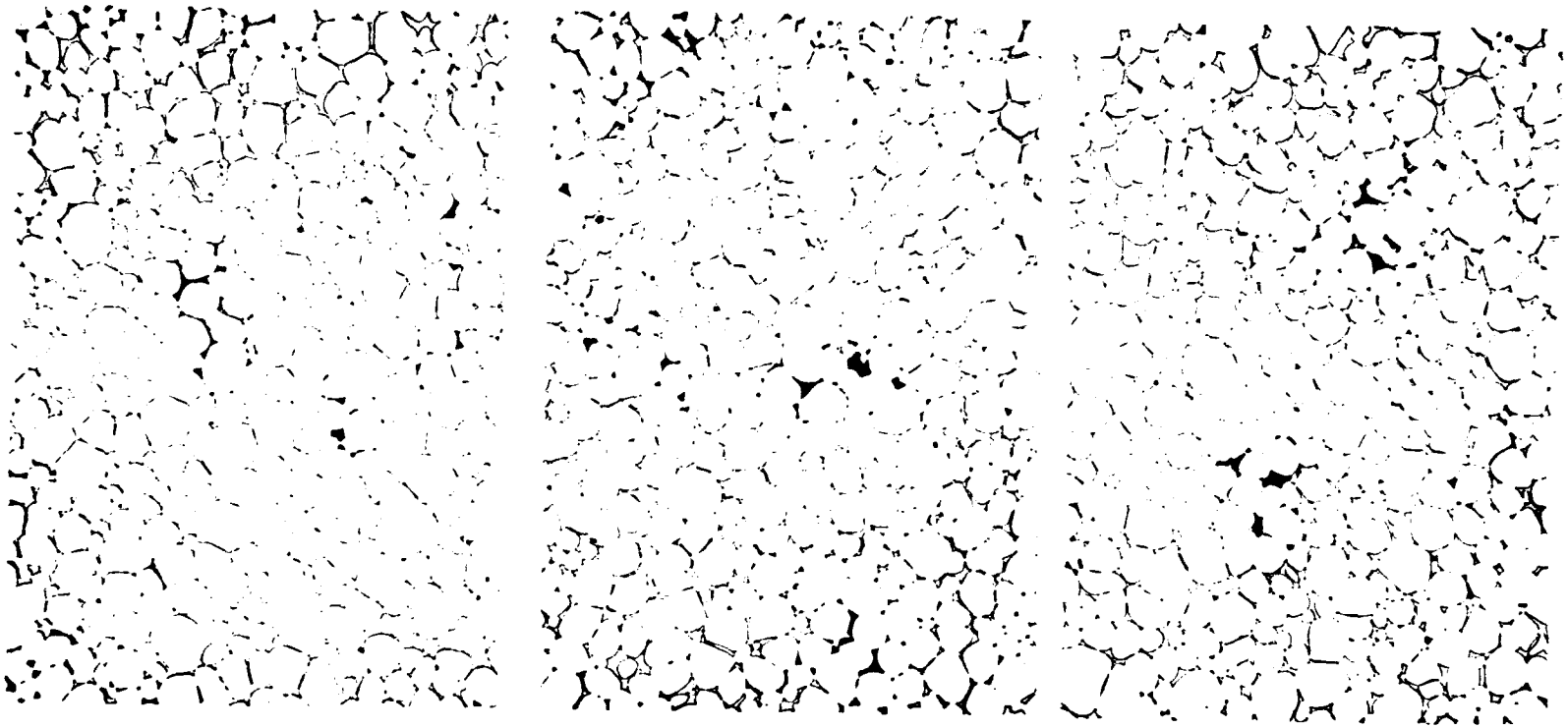


Figure 15. Light Micrographs, 100X, from Test 45

DISCUSSION OF RESULTS

In discussing the 751 alloy (97.1W-1.6Ni-0.7Fe-0.5Cu-0.1Co) and the results of our work, the most important consideration is the role the copper plays. Without copper density is easy to achieve as well as low oxygen levels and high elongations. All are problems with the copper-containing alloys which appear to be gas related. The pores in the low density pieces are rounded indicating a gas bubble as opposed to areas where matrix never entered. during solidification.

One possible source of trapped gas in the copper is hydrogen diffusing through the copper and reacting with oxygen to form water. The water molecule in turn can't easily diffuse out.

A. Density

In solid state sintering we expect higher densities as the particle size decreases. We didn't expect to see this, however, in the liquid phase sintering of the 751 alloy. Because of the enormous growth that the tungsten particles experience during sintering (>10X), it doesn't seem that starting particle size would be all that important. Our experience with WN-107 was that coarser particle sizes gave slightly better densities.

A possible explanation is that with finer tungsten particle sizes the higher surface to volume ratio would yield lower oxygen levels in the tungsten prior to the melting of the copper. Surface oxygen which is higher in fine tungsten particles would be removed during the low temperature holds.

The necessity of using slow ramps between 1000 °C and the sintering temperature to achieve good densities in large diameter bars must be related to the low melting point of the copper. After it melts at 1083 °C,

it continues to form new composition in equilibrium with the temperature. In the nickel-iron system when the nickel melts (1450 °C), all the iron will dissolve forming a composition that remains liquid because the eutectic occurs at about the same temperature as the melting point of nickel. In small bars of the 751 alloy we got better densities when the copper, cobalt, and nickel were prealloyed.

Another possible reason for difficulty in obtaining good density when copper is present may be related to the shape of the tungsten grain. In alloys with copper the grains appear squared with more straight sides. This makes long narrow channels which may hinder the removal of porosity. Many times we found a pore in the center of a tungsten grain caused apparently by tungsten grains coalescing around a pore.

We think that the crack and ring porosity we saw are manifestations of the general porosity problem with the 751 alloy.

It appears that these defects result as gas is rejected from the matrix during solidification. Because no cracking occurred during a nitrogen run, the gas must be hydrogen or water.

B. Tensile Properties

From our results it is clear that elongation is related to oxygen levels. Figure 16 is a plot of oxygen values versus the elongation. Below 20 ppm of oxygen there appears to be an inverse relation of tensile elongation to oxygen. We suspect that if we could have obtained low oxygen levels in the 751 alloy we would have obtained tensile properties similar to the 901 alloy. Some have suggested that the increased elongation with low oxygen levels is due to a purification of the tungsten grains. The purer tungsten grains can be elongated more before failure. We agree with this because once a bar is properly sintered fracture appears to be dictated by when the tungsten grains fail. In poorly sintered bars the fracture occurs due to the separation of the matrix and tungsten particles.

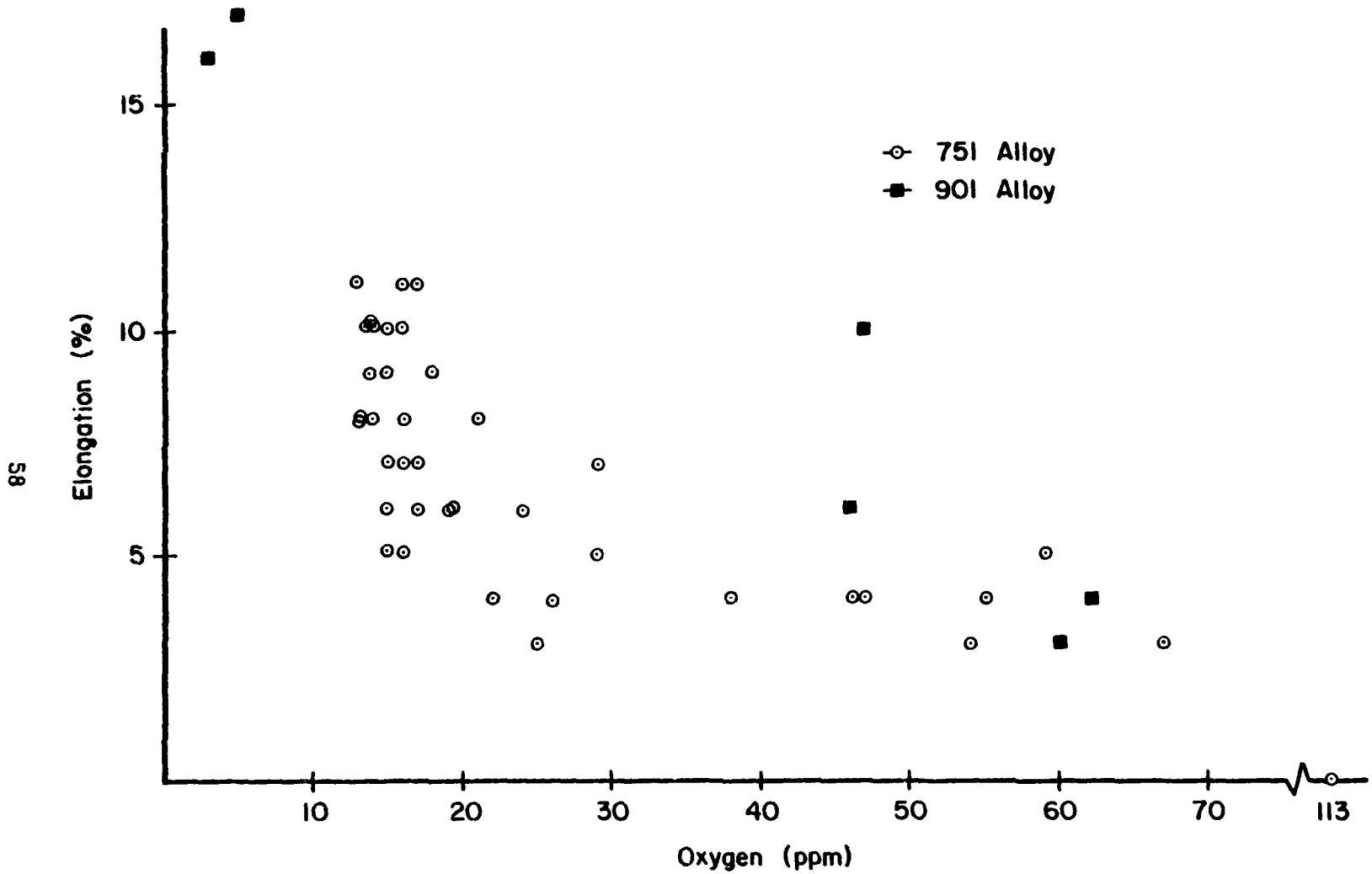


Figure 16. Room-temperature elongation versus oxygen content

What is difficult to explain is why it is so hard to obtain low oxygen levels in copper-containing heavy alloys when in similar alloys with no copper low oxygen levels are easy to obtain. We think the problem is related to the wider melting and solidification range that the copper causes. This is why slow heating and cooling rates must be used.

C. Uniformity of Properties

The properties we measured on the large bars (1.35" diameter) appeared to be very uniform. We believe this is because of the close control used during processing. Using a batch-type furnace like the Brew for sintering certainly aided in uniformity. Had we used a tube or muffle furnace, we suspect uniformity wouldn't have been as good. Ironically, we feel that we might have developed better tensile properties in a muffle furnace because of directional cooling.

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Army Materials and Mechanics Research Center
Watertown, Massachusetts 02172
TUNGSTEN ALLOY FABRICATION OF PENETRATOR MATERIALS
James R. Spencer
GTE Products Incorporated
Chemical and Metallurgical Division
Towanda, Pennsylvania 18848

Technical Report AMMRC TR 81-40, August, 1981, 65 pp.
illus - tables, Contract DAAG46-78-C-0009
Final Report, March, 1978 - May, 1980

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Sintering Temperature
Mechanical Properties
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Powder Metallurgy

Tungsten heavy alloy bars 1.35 inch in diameter were prepared and evaluated for uniformity of properties. Alloys contained 97.1% to 97.3% tungsten with primary emphasis on the 97.1W-1.6Ni-0.7Fe-0.5Cu-0.1Co composition. To achieve high uniform densities with this alloy, it was necessary to use fine tungsten powder (~1.5 μm) and slow heating rates between 1000 °C and the sintering temperature. This was not necessary with similar alloys containing no copper. Tensile elongation was found to be strongly dependent on oxygen levels in the sintered bars. Low oxygen levels gave the best results with 16-17% elongation when oxygen levels were below 6 ppm. These very low oxygen levels were only achieved with alloys containing no copper.

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